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Determination of Sodium at the Parts-Per-Trillion Level in the Presence of High Concentrations of Ethanolamine in Power Plant Waters

INTRODUCTION

The buildup of impurities at sub- $\mu\text{g/L}$ levels can result in their accumulation in steam generators or turbines through concentration mechanisms. Therefore, monitoring the presence of ionic impurities in cooling waters, boiler waters, feed waters, and steam condensate in both fossil-fueled and nuclear power plants is of critical importance. These impurities can propagate stress corrosion cracking and other corrosion mechanisms in turbines, steam generator tubing, and other plant components. This corrosion can eventually lead to component failures and plant shutdowns, resulting in millions of dollars of lost revenue.¹ Corrosive ions, such as sodium, chloride, and sulfate, can be minimized by continuously monitoring their respective levels and maintaining these levels as low as possible by taking the appropriate action.

The measurement of ultratrace levels of sodium and trending of this measurement provides valuable information for preventing corrosive conditions, unacceptable contamination levels, and other depositing conditions. The analysis of sodium can be applied to boiler systems to detect condenser leaks, breakthroughs in cation exchangers, condensate polisher failures, steam purity for turbine use, and assurance that no excess carryover is occurring from the process. Sodium content is also monitored in treated boiler water and the

condensate-containing additives that are needed for pH adjustments or oxygen control. In the past, morpholine was used as an additive in power plant waters because it was less volatile than the commonly used ammonia under all volatile treatment (AVT).² The steam condensate's pH from the turbine cycle could be increased to a level that significantly reduced flow-assisted corrosion, which was possible due to the lower volatility of morpholine. However, ammonia was still present from the thermal decomposition of morpholine. Less volatile amines with higher base strengths, such as ethanolamine, eventually replaced morpholine.³

As the acceptable limits of ionic impurities in the power industry have become more stringent, the requirements for the measurement technique have become more rigid to ensure the water quality is within acceptable operating parameters. The accurate and reliable determination of sodium at the sub- $\mu\text{g/L}$ levels in the presence of ethanolamine, typically in the thousands of $\mu\text{g/L}$ level, is a challenging analytical problem. In-line sodium analyzers are not reliable at the low- to sub- $\mu\text{g/L}$ levels and therefore have not gained the confidence of many users in the power industry.¹ Furthermore, sodium analyzers are incapable of detecting sodium at the industry levels required to prevent corrosive contamination.⁴ Ion chromatography (IC) has become an essential technique for addressing these challenges because IC can determine sub- $\mu\text{g/L}$ ionic impurities in power plant waters.¹⁻⁸

Over the past 20 years, IC has undergone many advances that have enabled the determination of cations at the low- to sub- $\mu\text{g/L}$ concentrations. More recent advances have further improved trace cation analysis, even in the presence of high concentrations of other cations. The first improvement was the development of the IonPac[®] CS16 column, a unique hydrophilic, high-capacity, carboxylated, functionalized cation exchanger that provides high resolution and peak efficiencies for the separation of common inorganic cations and amines.⁹ Typically, methanesulfonic acid (MSA) eluents are used with the IonPac CS16 column in either isocratic or gradient mode in conjunction with suppressed chromatography, resulting in sensitive detection for cations at trace levels. Another recent advance in IC is the development of on-line electrolytic generation of MSA using the EG40 module.¹⁰ This module allows continuous uninterrupted system operation and eliminates user-induced eluent preparation errors by providing in-line, contaminant-free eluents. Unlike bottled eluent systems, the use of the EG40 module reduces the probability of introducing contaminants in a trace system. The above benefits are particularly important for continuously operated systems such as power plants. The introduction of the Atlas[®] electrolytic suppressor represents another advance in IC.¹¹ This suppressor improves the determination of cations at trace levels by providing low baseline noise and fast equilibration times. The most recent advance in IC is the introduction of a new continuously regenerated cation trap column (CR-CTC).¹² The CR-CTC improves trace analysis by further purifying the electrolytically generated eluent and significantly reducing background conductivity. Unlike conventional trap columns that require frequent off-line chemical regeneration, the CR-CTC is a continuously electrolytically in-line-regenerated column. This benefit is particularly attractive for trace analysis because it translates into no downtime. Additionally, this in-line-regenerated column eliminates the possibility of introducing contaminants from the chemicals used in off-line regeneration.

In this application note, we describe an IC method that incorporates the most recent advances for trace sodium determination in power plant matrices. This method includes a sample preconcentration column to allow for the quantification of sodium at the ng/L levels. The analytes are separated on a new high-capacity cation exchanger using on-line electrically generated MSA and detected using suppressed conductivity. The method's performance is evaluated by analyzing synthetic samples containing high concentrations of ethanolamine that are representative of samples encountered in the power industry.

EQUIPMENT

Dionex DX-600 ion chromatography system consisting of:

- GP50 gradient pump with vacuum degas option
- ED50A Electrochemical Detector with conductivity cell and DS3 Detector Stabilizer
- LC30 Chromatography Enclosure equipped with a Rheodyne Model 9126 injector, PEEK, rear-loading (Dionex P/N 052291)
- EG40 Eluent Generator
- EluGen[®] EGC-MSA cartridge (Dionex P/N 053922)
- CR-CTC (Dionex P/N 060478)
- EG40 CR-TC Add-on Kit (Dionex P/N 060476)
- Pressurized Sample Vessel (Dionex P/N 037460)
- Dionex DXP Single-Piston Pump (Dionex P/N 043047)
- Chromeleon[®] Chromatography Workstation

REAGENTS AND STANDARDS

Deionized water, Type I reagent-grade, 18 M Ω -cm resistance or better

Sodium Chloride, reagent-grade (J.T. Baker, Inc.)

Ethanolamine, 99%, reagent-grade (Aldrich Chemical Co.)

CONDITIONS

Columns:	IonPac CS16 Analytical, 3 × 250 mm (Dionex P/N 059596) IonPac CG16 Guard, 3 × 50 mm (Dionex P/N 079931)
Eluent:	20 mM MSA
Eluent Source:	EG40 with CR-CTC
Eluent Flow Rate:	0.5 mL/min
Temperature:	60 °C
DXP Flow Rate:	2 mL/min
Sample Volume:	10 mL
Concentrator	
Column:	IonPac CG16, 5 × 50 mm (Dionex P/N 057574)
Detection:	Suppressed conductivity, CAES™ (Dionex P/N 056118), placed outside the LC30; power setting, 34 mA
Expected	
Background:	<0.3 µS
Expected Noise:	~0.5 nS
Expected System	
Backpressure:	2400–2600 psi
Run Time:	20 min

PREPARATION OF SOLUTIONS AND REAGENTS

1000 mg/L Sodium Standard Solution

Dissolve 2.541 g of sodium chloride in 1000 mL of deionized water. Working standards were prepared by serial dilutions from the 1000 mg/L (ppm) concentrate. Working standards containing less than 1 mg/L were prepared every 2–3 days, and standards containing less than 1 µg/L were prepared daily and analyzed almost immediately after preparation.

1000 mg/L Ethanolamine Standard Solution

Dissolve 1 g of ethanolamine in 1000 mL of deionized water.

Matrix Preparation

In this study, three different matrices were prepared containing 3, 5, and 10 ppm ethanolamine. Each matrix was spiked with ~0.025 µg/L sodium.

SYSTEM PREPARATION AND SETUP

Prepare the Cation Atlas Electrolytic Suppressor (CAES) for use by hydrating the membranes using either a 3- or 5-mL syringe to push approximately 3 mL of deionized water through the “Eluent In” and “Regen In” ports of the suppressor, respectively. Allow the CAES to stand for at least 20 min before installing in the IC system. For more information on CAES operation, consult the *CAES Quickstart Guide* (Document No. 031771).

Install the EGC-MSA EluGen cartridge according to the instructions in the operator’s manual for the EG40 Eluent Generator System (Document No. 031373). Install the CR-CTC between the outlet of the EGC-MSA and the inlet of the EG40 degas device. Figure 1 illustrates the system diagram, showing the plumbing of the CR-CTC. For more information on installing the CR-CTC, consult the *CR-CTC Manual*. This configuration effectively eliminates potential contaminants from the water stream leading to the EGC.

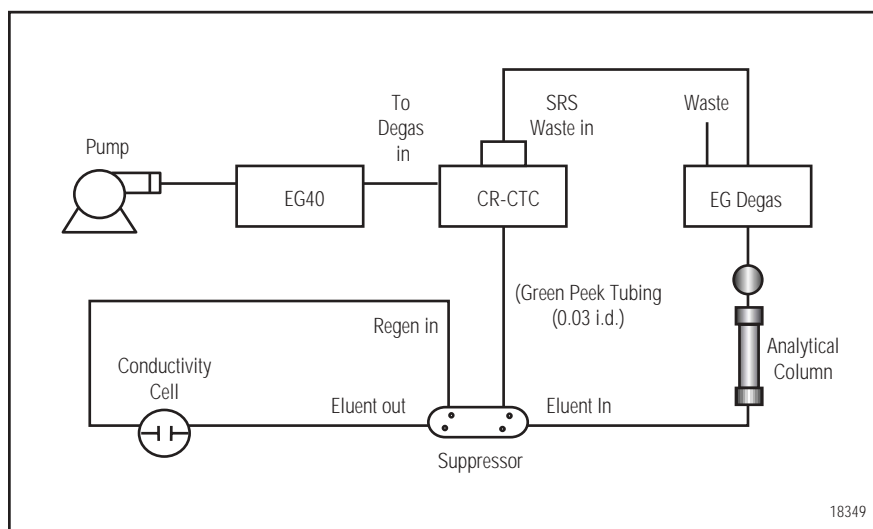


Figure 1. Ion chromatography system with CR-CTC configuration.

Configure the preconcentration system according to the diagram shown in Figure 2. To minimize system void volume and improve column operation, 0.005-in. (0.125-mm) tubing should be used for connecting the columns and suppressor. The tubing lengths should be kept as short as possible and the ends cut smooth and straight to eliminate irregularity in flow and unwanted dead volume. To maintain efficient operation of the DXP, the sample or standard feeding this pump should be pressurized with 5 psi (34 kPa) of helium. For this purpose, and to minimize contamination from the environment, we strongly recommend using a pressurized vessel to enclose the standard or sample for this method.

Install a 3 × 50 mm IonPac CG16, 3 × 250 mm IonPac CG16 column, and the CAES. Be sure the CAES is placed outside the LC30 before setting the temperature to 60 °C. Begin flow of the gradient pump at 0.5 mL/min. If the system backpressure is less than 2000 psi, then additional PEEK tubing (0.003-in.) should be added between the EG40 degas device and the inlet of the injection valve. The optimal total system pressure should be between 2400 to 2600 psi. Ensure the backpressure does not exceed 3000 psi. Note that when the valve is in the inject position, ~200 psi increase in backpressure will result from the 5 × 50 mm IonPac CG16 Concentrator Column. Confirm that there are no leaks in the chromatographic pathway.

To begin, set the EG40 to deliver 20 mM MSA at 0.5 mL/min and set the current of the CAES to the

recommended current setting of 34 mA. Allow the LC30 oven to stabilize at an operating temperature of 60 °C. Note that increasing the temperature will result in a decrease in the eluent viscosity, causing a decrease in the system pressure. Make sure the system pressure is at least 2000 psi at 60 °C when the valve is in the fill position. Assess the stability of the baseline and peak-to-peak noise by measuring a deionized water blank. In a representative 1-min segment of the chromatogram, the peak-to-peak noise measurement should be less than 1 nS. We strongly recommend equilibrating the system by running under the method conditions overnight prior to performing trace analysis. Shutting off the system after use each day is not recommended because this may result in brief changes in the background conductivity and an increase in baseline noise. Therefore, for optimal performance in a trace-level analysis system, we recommend running the IC continuously. Be sure there is an adequate supply of DI water to feed the EG40 for unattended operation.

RESULTS AND DISCUSSION

General Precautions

The most obvious concern when performing trace analysis is the enormous potential of contamination. Therefore, extreme caution should be taken throughout the entire process to eliminate or minimize potential contaminants. Disposable, powder-free PVC gloves should be worn when working on the IC system,

standards, samples, or blanks. Because even gloves can potentially leach ions, extra caution should be taken to minimize contact with all solutions. Prior knowledge of the IC system's history should be known, because systems previously used for analysis of a high concentration of anions (the counter ion of most anions is sodium) and cations will result in additional time required to remove the residual ions prior to achieving a contaminant-free system. Nonetheless, the entire flow path of the chromatographic instrumentation (e.g., eluent bottle of DI water to supply the EG40,

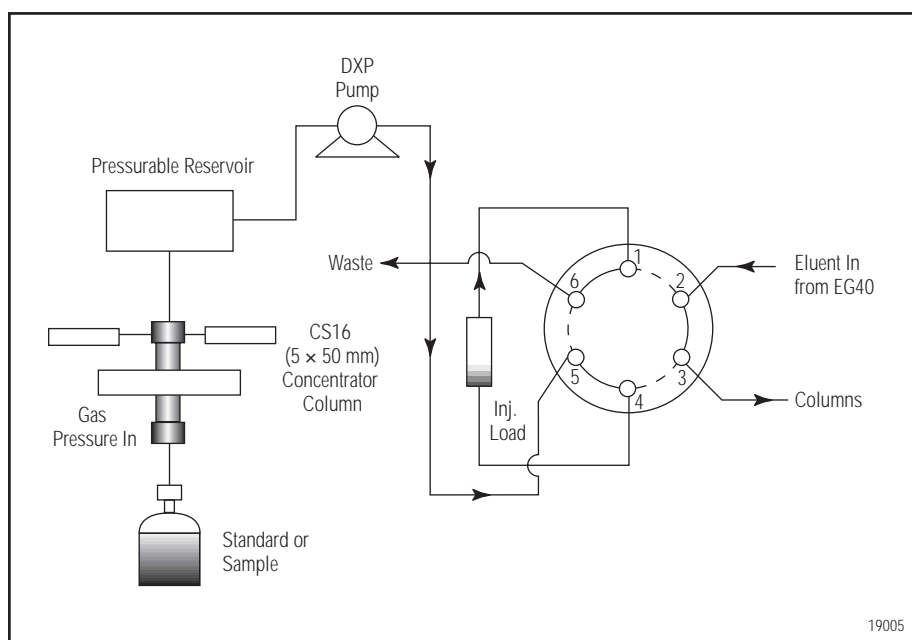


Figure 2. Ion chromatography preconcentration system.

pump, tubing, injection valve, columns, suppressor, and conductivity cell) represents potential sources of contamination. The CR-CTC is designed to effectively eliminate the system cation contaminants that may result from some of the sources previously listed.

Containers for Blanks and Standards

The best containers were found to be Corning polystyrene tissue-culture flasks with plug seals. The containers were prepared by rinsing each flask and lid three to four times with ultrapure water until overflowing. The flask was then completely filled to the top, capped tightly, and allowed to soak overnight. This process was repeated until an acceptable and consistent blank was achieved. Be sure not to overtighten the lids; check that the lids are well threaded, otherwise the lids may tear at the seams. These containers were used for blanks, standards containing less than 1 mg/L sodium, and simulated matrices. Prior to preparing a standard in a soaked and rinsed flask, a blank was measured from the flask that would contain that particular standard. Each flask was dedicated to specific concentrations and was clearly marked to distinguish it from other containers. When a flask was not in use, it was always kept full with DI water, and rinsed and soaked periodically.

Loading of the CG16 (5 × 50 mm) Concentrator Column

The use of an autosampler for loading samples and standards was avoided because the high backpressure of the concentrator was too much for the autosampler to displace the liquid into the concentrator column. In this method, a DXP pump was chosen for delivery of all samples and standards for several reasons: (1) the DXP can tolerate pressures up to 4000 psi; (2) the pump can be set to operate at high flow rates (up to 4 mL/min) to minimize loading time; and (3) a pressurized reservoir may be used to isolate the sample from the environment and therefore minimize the possibility of contamination during the sample loading process. Prior to setting the DXP flow rate, the check valves should be soaked in DI water. After reassembling the pump (wearing powder-free PVC gloves), set the DXP flow rate to 2 mL/min through the CG16 (5 × 50 mm). After collecting for a predetermined time, weigh the amount of liquid in a tared container to verify the DXP flow rate. If an inconsistent flow rate is observed, then we recommend

loosening the check valves to remove trapped air bubbles. The flow rate should be verified periodically to ensure consistent flow throughout the method. The pump was rinsed with DI water for at least an 8-h period, but may require additional rinsing before the blank reaches an acceptable and stable level.

Tefzel tubing was attached to the inlet of the pressurized reservoir and PEEK tubing (0.030-in.) extended from the outlet. The Tefzel tubing was always kept in DI water, housed in a soaked and rinsed polystyrene container within the pressurized reservoir, regardless of whether the pump was in use. A separate polystyrene container, filled with DI water, was placed on the outside of the reservoir and used to place the Tefzel tubing in cases where the containers had to be changed from the reservoir. This container was dedicated for this use only, and rinsed and soaked with DI water periodically. When removing the cap from the reservoir, be sure to prevent the Tefzel tubing from contacting any surfaces, because such contact will lead to contamination.

Breakthrough Volume of CG16 (5 × 50 mm) Concentrator Column

When a sample containing a high ionic strength is preconcentrated, the sample can act as an eluent and prevent the retention of the analytes of interest on the concentrator column.¹³ Because the resins in concentrator columns have a finite capacity (e.g., the column can retain a given amount of ions from a sample matrix), the target analytes are no longer retained if the capacity of the column is exceeded. The volume of sample where the capacity is exceeded is called the breakthrough volume. In this method, ultratrace levels of sodium were measured in the presence of high concentrations of ethanolamine. Because the amine is present at significantly higher concentrations than the target analyte, the sample volume concentrated is limited by the breakthrough volume. For a sample containing 0.250 µg/L sodium in the presence of 5000 µg/L ethanolamine, the breakthrough volume of the CG16 (5 × 50 mm) was predetermined to occur at a sample volume of ~30 mL. Of course, this breakthrough volume will vary depending on the amount of ethanolamine in the sample. For more information on determining the breakthrough volume, consult Dionex Technical Note 8.

System Optimization

In the power industry, trace levels of sodium are often measured in the presence of large amounts of ethanolamine, ammonium (as a breakdown of ethanolamine), and possibly other amines. This presence causes an analytical challenge because the large amounts of amine could potentially overload the concentrator and analytical column, resulting in a difficult separation and quantification of sodium at the parts-per-trillion (ppt) level. In this study, the IonPac CS16 was chosen as the analytical column because its high ion-exchange capacity of 8400 $\mu\text{eq}/\text{column}$ (5-mm format) or 3000 $\mu\text{eq}/\text{column}$ (3-mm format) is ideal for resolving high concentration ratios of sodium and ethanolamine. This high capacity is achieved by using a smaller bead diameter size (5.5- μm), a higher density of grafted carboxylated cation-exchange sites, and a larger column format. For accurate determinations of sodium at the sub- $\mu\text{g}/\text{L}$ level in the presence of high amounts of ethanolamine, it is necessary to maximize the peak response, while not overloading the capacity of the concentrator column, and still maintain good resolution between the other analytes present in the matrix. To enhance the sodium response, we used an IonPac CG16 (5 \times 50 mm) as the concentrator column. This 5-mm guard column was chosen because it provides nearly three times the exchange capacity of the 3-mm format. Therefore, it provides the maximum loading capacity possible for matrices typically encountered in the power industry, while still producing the resolution and peak efficiencies needed to determine sodium at the ppt level. For the analytical separation, the 3-mm format was chosen over the larger-bore (5-mm i.d.) format to obtain the advantages of (1) lower eluent flow rates, (2) improved peak shapes, and (3) reduced loading times (e.g., lower concentration volumes required for an equivalent analyte response, necessary for the 5-mm format).

When the chromatographic baseline noise is minimized, analyte response improves and the detection limit is lowered. This result was accomplished using a CAES. One of the main advantages of the CAES is it provides low baseline noise (typically 0.2–0.5 nS/cm for cation analyses), even when operated in the AutoSuppression[®] recycle mode. The maximum suppression capacity of the CAES is a 25 mN eluent at ≤ 1.0 mL/min. When the suppressor was supplied with 34 mA of current and operated in the recycle mode, the peak-to-peak noise measured in a 1-min representative

segment of the chromatogram was less than 0.5 nS/cm.

Analyte response may also be increased by operating the column at elevated temperatures, which can often improve chromatographic efficiencies, enhance resolution, and decrease retention times. At higher temperatures, the carboxylic acid groups on the resin are less ionized and therefore decrease the effective column capacity, resulting in shorter retention times and improved sensitivity.¹⁴ In this method, the column was operated at 60 °C to (1) decrease retention times for improved analyte response at the ng/L level, (2) improve peak efficiencies for the high amounts of ethanolamine present to decrease overall run times and increase sample throughput, and (3) improve resolution between sodium and an unknown peak present in the simulated sample matrix.

It is important to establish a system blank and ensure the stability of the blank analyses over a period of several days. Establishing an acceptable blank level may take several weeks. However, this time will depend on the cleanliness of the laboratory, purity of the water source, presence of contaminants in the IC system, and other factors that can contribute to high blank levels. In this study, the blank was analyzed over a four-day period, resulting in an average sodium concentration of

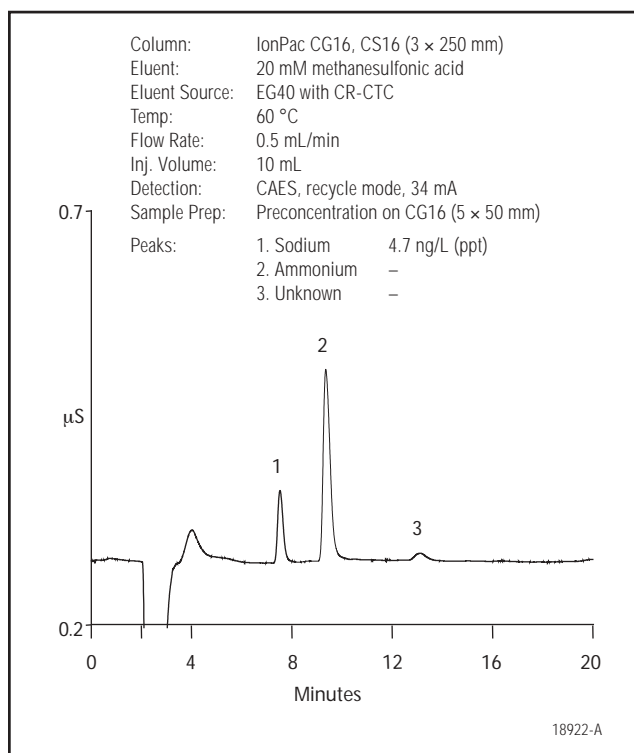


Figure 3. A representative blank chromatogram of deionized water.

4.5 ± 0.7 ng/L (n = 21). Figure 3 shows a representative blank chromatogram of deionized water. Trace amounts of sodium, ammonium, and an unknown peak from the deionized water were detected.

Method Performance

The calibration curve for sodium was obtained by preparing standards in deionized water. Table 1 shows the calibration linearity and method detection limits (MDLs) for sodium. The MDL was determined by performing seven replicate injections of 25 ng/L sodium, fortified in high-purity reagent water. The MDL was calculated using the standard deviation from seven replicate injections and multiplying by the Student's *t* value for a 99% confidence level. The result was a calculated MDL of 3.2 ng/L using a 10-mL sample volume. The precision of the retention times, based on

Table 1. Calibration Linearity and MDL for Sodium Using a 10-mL Sample Volume

Analyte	Range ^a (ng/L)	Linearity (r ²)	Calculated MDL ^b (ng/L)	MDL Standard (ng/L)
Sodium	25.0–250	0.9990	3.20	25.0

^aCalibration levels were 25, 50, 100, and 250 ng/L (each standard injected in duplicate)

^bMDL = *t* × *S* where *t* = Student's *t* value for a 99% confidence level and a standard deviation estimate with *n* – 1 degrees of freedom (*t* = 3.14 for seven replicates of the MDL standard), and *S* = standard deviation of the replicate analysis

the replicate injections, yielded an RSD of 0.33%. Figure 4 shows a representative chromatogram of 25 ng/L sodium prepared in deionized water.

The method performance was evaluated by analyzing synthetic samples containing ethanolamine concentrations ranging from 3 to 10 mg/L that simulate samples encountered in the power industry. Figure 5 shows a chromatogram of 3 mg/L ethanolamine with no additional sodium. The average concentration of sodium found in the matrix, based on triplicate injections, was 5.5 ng/L, indicating that no additional sodium was added from the matrix. As this figure illustrates, an unknown peak eluted between sodium and ethanolamine. However, due to the high peak efficiencies resulting from the elevated column temperature, the unknown peak does not interfere with the determination of sodium, even at the ppt levels. Figure 6 shows a

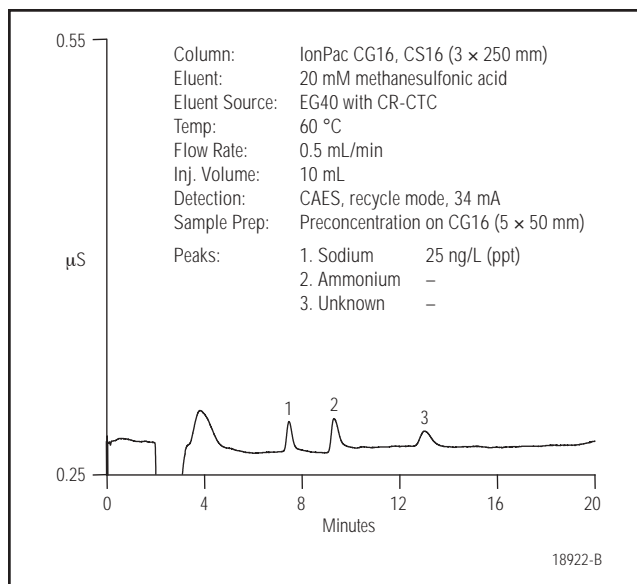


Figure 4. A representative chromatogram of deionized water spiked with 25 ng/L sodium using a 10-mL sample volume.

chromatogram obtained by spiking the sample with 22 ng/L sodium. The average spiked recoveries for sodium based on seven replicate injections of the 3-mg/L ethanolamine sample was 99%.

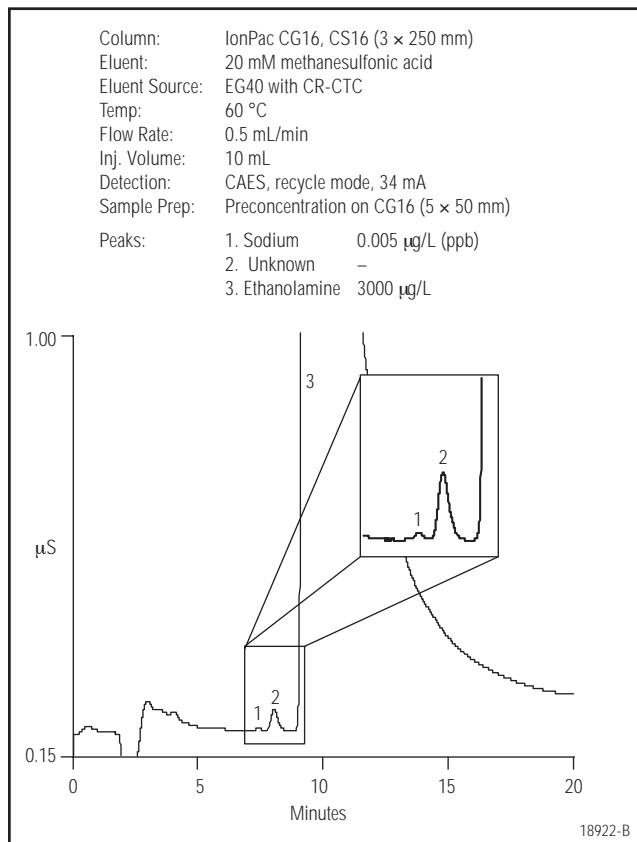


Figure 5. A representative chromatogram of 3000 µg/L ethanolamine.

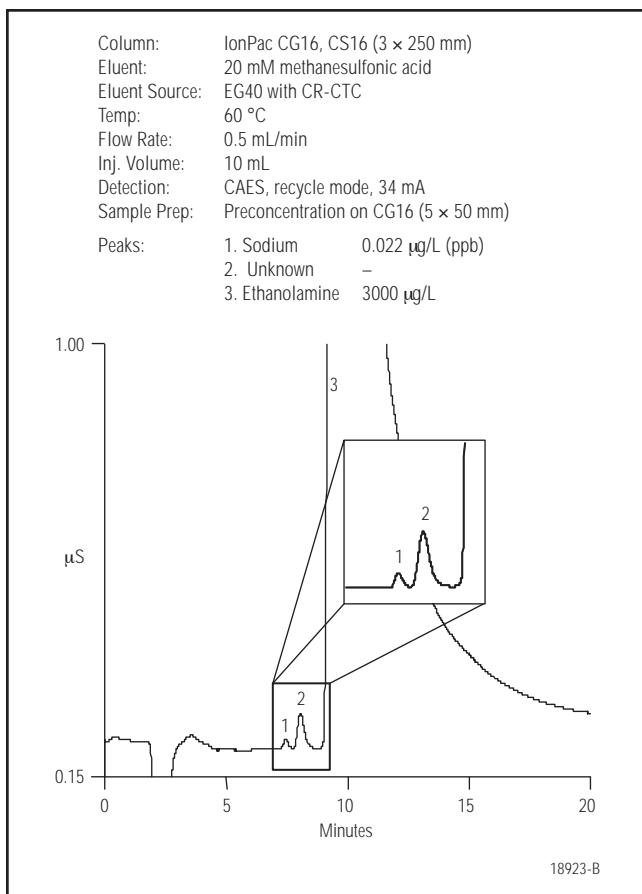


Figure 6. A representative chromatogram of 3000 µg/L ethanolamine spiked with 0.025 µg/L sodium.

Table 2 summarizes the data for the spiked recoveries of sodium in matrices containing 3–10 mg/L ethanolamine. As shown, the average recoveries ranged from 85 to 110%. This range indicates that the method performed well for samples spiked with low ppt sodium and that the high amounts of ethanolamine do not significantly influence the separation and quantitation of sodium at the target levels. The retention time precision data for sodium separated on the IonPac CS16 was also evaluated by performing replicate analyses of ~25 ng/L sodium in deionized water and the synthetic samples summarized in Table 3. As shown, the relative standard deviations (RSDs) ranged from 0.06 to 0.33%, indicating the retention time of sodium was independent of the amount of ethanolamine present.

Ethanolamine Concentration (µg/L)	Spiked Sodium Concentration (µg/L)	Average* Sodium Recovery (%)	RSD* (%)
0.022	3000	99.0	3.68
0.025	5000	109.7	2.78
0.027	10,000	85.0	6.89

* The average recovery and relative standard deviations (RSDs) were calculated from seven replicate injections (n = 7).

Ethanolamine Concentration (µg/L)	Average* Sodium Retention Time (min)	RSD* (%)
0	7.46	0.33
3000	7.42	0.11
5000	7.41	0.06
10,000	7.41	0.13

* The average recovery and relative standard deviations (RSDs) were calculated from seven replicate injections (n = 7).

CONCLUSION

A method based on a high-capacity, carboxylated, cation-exchange column was developed for the isocratic separation of ultratrace levels of sodium in a matrix containing high concentrations of ethanolamine in 20 min. The column provided good resolution and peak efficiencies, even in matrices of disparate concentration ratios of sodium to ethanolamine. A larger-diameter concentrator column of the same material as the analytical column provided higher loading capacities for the high-ionic-strength samples examined in this study, allowing for the detection and quantitation of sodium down to the low-ng/L levels. Excellent linearity for sodium was obtained at the target levels, resulting in a correlation coefficient of 0.999. The precision based on multiple injections of the same sample was less than 10%, even in the presence of 10,000 µg/L ethanolamine. Method detection limits at the single-digit ng/L level may be achieved by following the necessary precautions listed in this application note.

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SUPPLIERS

- Aldrich Chemical Co., P.O. Box 2060, Milwaukee, WI 53201 USA, Tel: 800-558-9160, www.aldrich.sial.com.
- J. T. Baker, 222 Red School Lane, Phillipsburg, NJ 08865 USA, Tel: 800-582-2537, www.jtbaker.com (order from VWR at www.vwrsp.com).



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